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Determination of ^{90}Sr, ^{14}C, ^{40}K, ^{134}Cs, and ^{137}Cs Activities in the Total Diet Around Potential Nuclear Power Plant Sites in Turkey

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Abstract This paper presents the results of a search for determining the activity concentrations of ^{90}Sr, ^{14}C, ^{40}K, ^{134}Cs and ^{137}Cs in mixed diet samples by using liquid scintillation and gamma spectrometric methods. Samples were collected by using duplicate portion sampling technique and dried by freeze dryer after their homogenization process. The method used in the ^{90}Sr analysis is based on the separation of the daughter ^{90}Y at equilibrium with 10% di(2-ethyl-hexyl)phosphoric acid (HDEPH) in toluene and Cherenkov counting by a liquid scintillation spectrometer (LSS). ^{14}C activity concentration is determined by the combustion of the sample in an oxygen atmosphere to $^{14}CO_2$, absorption of $^{14}CO_2$ by special reagent, mixing with the scintillator and counting in liquid scintillation spectrometer. ^{40}K, ^{134}Cs and ^{137}Cs activity concentrations were measured by high purity germanium detectors in gamma spectrometer. An annual effective dose caused by these radionuclides was estimated using dose conversion coefficients and measured activity concentrations in samples.

Keywords Mixed Diet, ^{90}Sr, ^{14}C, ^{134}Cs, ^{137}Cs, ^{40}K, Cherenkov Counting, Liquid Scintillation Spectrometry, Gamma Spectrometry

Introduction

Humans are constantly exposed to a variety of sources of both natural and artificial radioactivity through inhalation, intake of food and water. The most important sources of unwanted artificial radionuclides in the environment are nuclear power plants in operation, atmospheric nuclear weapon tests and nuclear power plant accidents. Radionuclides in the environment contaminate food depending on the physical and chemical properties of soils and radionuclide uptake by specific plants [1]. These radionuclides are accumulated in plants through their roots and deposited on the stem and leaves through dust in the air. Therefore, contaminated soil becomes a source of radionuclides entering agricultural products, surface and subsurface waters for a certain time depending on the half-life and chemical and physical filtration in the soil. Inevitably some of them get into the food and water we ingest [2-3]. The ingestion of radiologically contaminated plants and animal products by humans contributes to the overall dose. Hence, the measurement of activity concentration of artificial radionuclides in diet such as ^{90}Sr, ^{14}C, ^{134}Cs and ^{137}Cs with the highest level of accuracy is very important in monitoring the food products especially following a nuclear power plant accident. In addition, determining the range of specific activity of radionuclides in the diet is necessary to be able to assess the risk of exposure [4].

According to EU Commision Recommendation of 8 June 2000 on the application of Article 36 of the Euratom Treaty concerning the monitoring of the levels of radioactivity in the environment for the purpose of assessing the exposure of the population as a whole (2000/473/Euratom), complete meals should be sampled to give a representative figure for the average level of radioactivity in mixed diet and, especially ^{90}Sr, ^{134}Cs and ^{137}Cs activity should be monitored [5]. The determination of the radionuclide concentration in the diet or individual food items constitutes an important element of an integrated program of radiological surveillance and assessment. Existing approaches such as the determination of dose from weighted average of the consumption of milk and milk products, leafy vegetables, grain products, other fruits and vegetables and meat usually are used to estimate the daily intake but such models do not take into account the culinary losses of radionuclides during the preparation of meals.

Turkey has no nuclear power plant in operation but, two four-unit plants are being built in Mersin and Sinop. As a result, measurement of the level of radioactivity in the local diet to determine the background is becoming a particularly urgent issue which is the main objective of this study. ^{90}Sr, ^{40}K, ^{134}Cs and ^{137}Cs analyses are routinely made in soil, food, water, milk and environmental samples in our laboratory. However, no comprehensive survey of the radionuclide activity concentration in the mixed diet has been conducted in these regions and no systematic work was done in Turkey in general. The findings will be used to strengthen and update monitoring programs, improve the assessment of doses to members of the public near nuclear sites, evaluate the actual radiation risks and to establish baseline information for future studies.

A project was proposed to TAEK (Turkish Atomic Energy Authority) for the assessment of radiation dose due to radionuclides in the human diet in potential nuclear power plant sites. The purpose of this project was to set up the needed infrastructure for the radioactivity analysis in human diet, install new systems, make up the deficiencies in existing methodologies, improve existing analytical methods for the determination of ^{14}C activity in mixed diet samples using liquid scintillation spectrometer and investigate the applicability of duplicate portion technique as the sampling procedure. The expected outcome is the increase in the number of radionuclides being analyzed in food sample matrices. In this context, this work describes the determination of the activity concentrations of ^{90}Sr, ^{14}C, ^{134}Cs, ^{137}Cs and ^{40}K in mixed diet collected by double portion sampling technique.

Experimental Work

Equipment

Ortec p-type high purity germanium detector with 110 % relative efficiency is utilized for the measurements of ^{134}Cs, ^{137}Cs and ^{40}K radionuclides. Canberra Genie2000 software was used to acquire and analyse the spectra. Activity concentrations of ^{90}Sr and ^{14}C isotopes were determined by using Perkin Elmer 1220 Quantulus ultra low-level liquid scintillation counter (Turku, Finland) with an external standard of ^{152}Eu which allows to measure external spectral quench parameter (SQP[E]). Analytical results were determined using WinQ and Wallac Easy View Spectrum analysis programs. Samples were homogenized using Waring® 4L laboratory blender and freeze-dried using Labconco® FreeZone 6L Benchtop Freeze Drying System. The dried samples were combusted with an automatic oxidizer (Model 307 by Perkin Elmer) into carbon dioxide ($^{14}CO_2$). All measurements were carried out with 20 mL Teflon coated low diffusion polyethylene vials (Perkin Elmer).

Reagents and Solutions

All chemicals were of analytical reagent grade. $^{90}Sr/^{90}Y$ (Cat. No. 7090; 3.711 $kBq.g^{-1}$) and ^{14}C (739.4 $Bq.g^{-1}$) certified reference sources for gamma and liquid scintillation counting were purchased from NIST and Eckert & Ziegler Isotope Products. The $^{90}Sr/^{90}Y$ certified solution was diluted in 1 M HCl in order to prepare solutions with a nominal activity of 0.338 $Bq.g^{-1}$. Bis(2-ethyl-hexyl)phosphoric acid (HDEHP) was purchased from Merck and 10 %(v/v) solution was prepared in toluene. Yttrium carrier (10 $mgY^{3+}.mL^{-1}$) was prepared by dissolving 1.27 g of Y_2O_3 in a minimum volume of conc. HNO_3 and diluted to 100 mL with deionized water. Quench curve determination, recovery and memory tests performed with the liquid ^{3}H ($1.57 \times 10^6 \pm 1.26$ % $dpm.g^{-1}$) and ^{14}C ($9.39\times10^5 \pm 0.94$ % $dpm.g^{-1}$) SpecChec standard solutions purchased from Perkin Elmer. Ultima Gold LLT, Permafluor E^+ scintillation cocktails and CarboSorb E trapping reagent (Perkin Elmer) were used.

Sample Preparation

The city of Mersin is located in the southeast part of Turkey and it has been selected as the pilot area, which is the one of the regions previously monitored for environmental radioactivity levels. The locations of the sampling points (Yanışlı, Aydıncık, Eceli, Gülnar and Sipahili) are shown in Figure 1. Samples were collected by 5 volunteeres who deposited a second portion identical to what they ate during the course of the meal into a 5L container for 3-consecutive days and saved in refrigerator.

The sampling group members were chosen to represent the local life-style and nutritional habits. The diet samples consisted primarily of regional grain, meat (red and white), vegetables, fruits, cheese, starch products and water. The amount of samples collected was between 6300-6800 g which have dry weights between 500-650 g. Samples were then homogenized using Waring® 4L laboratory blender and the whole homogenised samples were divided into small portions and freeze-dried using Labconco® FreeZone 6L Benchtop Freeze Dry System. A background sample was also collected from a volunteer staff in our laboratory, in order to validate the proposed methods used for the determination of ^{90}Sr and ^{14}C.

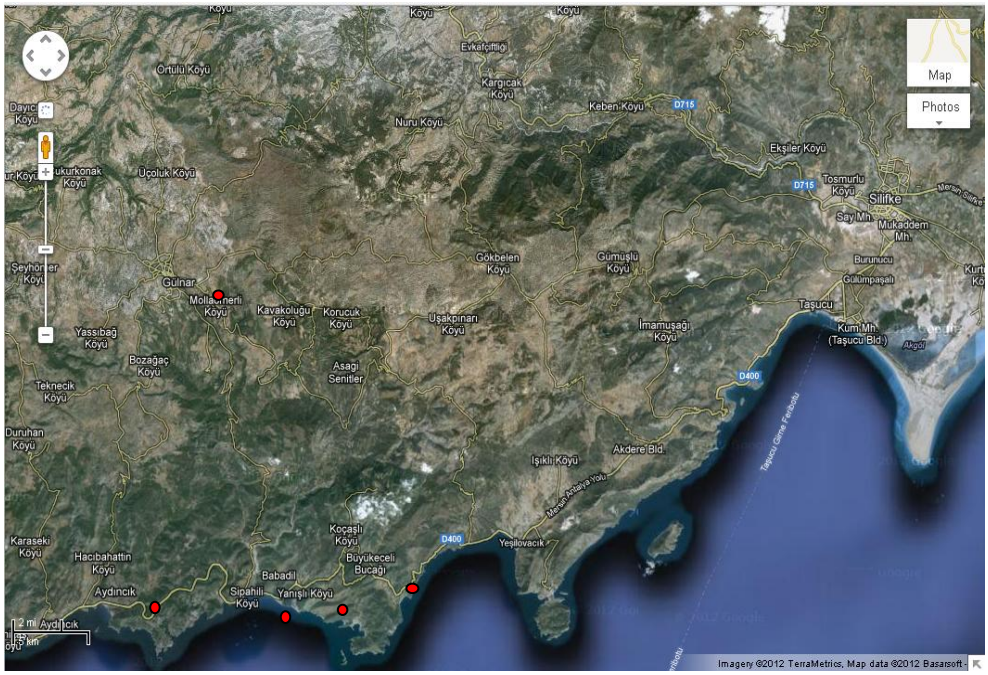

Figure 1: Map of Mersin with locations of the sampling points.

Measurement of ^{134}Cs, ^{137}Cs and ^{40}K

ISO 17025 accredited "Radioactivity analysis of ^{137}Cs and ^{134}Cs in foodstuffs" method was used to analyze representative portions of freeze-dried samples in gamma ray spectrometry laboratory [6]. The samples were filled in sample containers with standard geometry which was also used to construct the efficiency calibration of the detector. The calibration source is also a vegetable origin spiked material with a density of 0.55 g/cm^3. Sources were weighed with a balance having ± 17 mg uncertainty. The sample masses varied from 29 g to 31 g. The samples were counted for at least 60000 s. Since ^{137}Cs and ^{40}K does not require coincidence correction, only background, decay and self-absorption corrections were applied. The uncertainty component due to sample in homogeneity was included in the uncertainty budget along with the statistical, nuclear data, weighing, efficiency and self-attenuation correction uncertainties. ^{134}Cs activity concentration was below minimum detectable activity concentration (MDC) for all the samples.

Method validation

Spiked samples were studied prior to the real samples in order to set out clearly the procedure in the application of the liquid scintillation spectrometric method for the determination of ^{90}Sr and ^{14}C radioisotopes in diet samples [7-8]. For this purpose spiked samples were prepared by adding known amount of ^{90}Sr/^{90}Y (3.711 kBq.g^{-1}) and ^{14}C (739.1 Bq.g^{-1}) standard reference solutions. The quality parameters established on the validation of the liquid scintillation spectrometric method comprised radiochemical recovery, MDC, trueness (as relative error percentage) and repeatability (as relative standard deviation percentage, %RSD).

Measurement of ^{14}C

A portion of homogenized and freeze-dried diet samples were weighed in the combustion cups and spiked with standard ^{14}C solution (739.4 Bq.g^{-1}). The cups give off no residue in the combustion process and have no effect on the sample measurements. Sample sizes varied between 0.4346 and 0.7082 g dry weight. Three parallel samples were carried out for each diet sample. The combustion itself occurs in an oxygen flame and it takes 1-2 minutes per sample. The resulting product $^{14}CO_2$+$^{12}CO_2$ is then directly absorbed into the 10 mL of Carbo-Sorb E. After absorption of the $^{14}CO_2$, the instrument automatically adds 10 mL Permafluor E$^+$ to the sample. The absorber/scintillator ratio used in this study is recommended in the manufacturer guidelines according to the sample sizes. After combustion, samples were measured with low level background liquid scintillation spectrometer Quantulus 1220 by Perkin Elmer. The spectra of ^{14}C by LSS is shown in Figure 2.

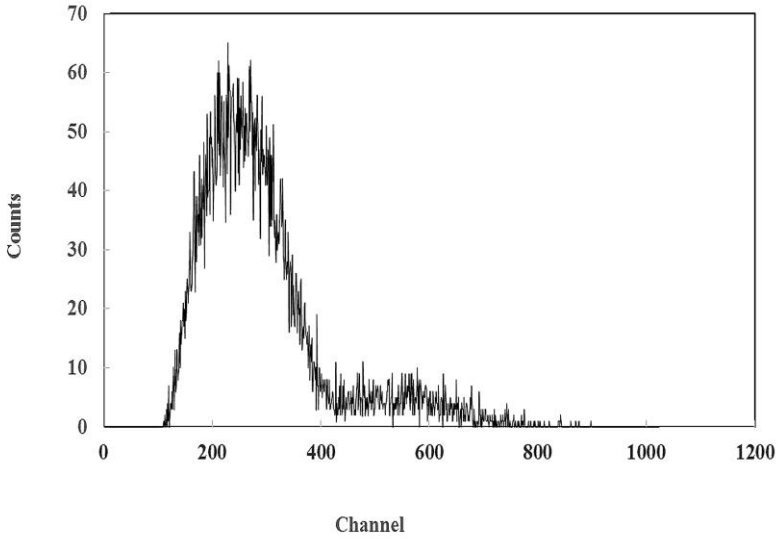

Figure 2: Spectrum of ^{14}C for spiked diet sample obtained from LSS.

A quench curve was constructed in order to determine the counting efficiency of each measurement. The curve was drawn by preparing 8 calibration samples with different amounts of CarbosorbE (2, 4, 6, 8, 9, 10 mL) and with the same amount of ^{14}C standard solution (78500 dpm). Each calibration sample was counted for 20 min. The counting efficiencies of the calibration samples were calculated and plotted against the external spectral quench parameter (SQP(E)) (Figure 3). The efficiencies for each sample were then calculated from the fitted curve using the SQP(E) value for each sample.

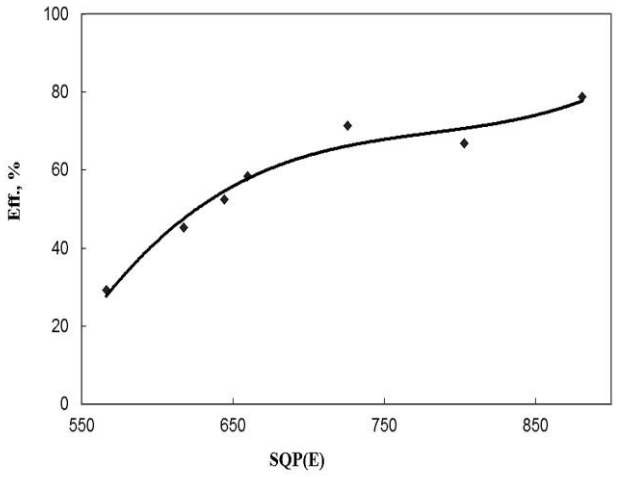

Figure 3: The quench curve of ^{14}C samples.

Measurement of ^{90}Y (^{90}Sr)

The method used for the ^{90}Sr analysis is based on the separation of the daughter yttrium-90 (^{90}Y) at sequliar equilibrium with bis(2-ethyl-hexyl)phosphoric acid (HDEPH) solvent extraction followed by counting Cherenkov radiation. A subsample of 20 g of blank diet sample was weighed into a porcelain capsule and spiked with NIST traceable ^{90}Sr standard solution having activity of 0.338 Bq/mL. The spiked samples were ashed by using Milestone microwave ash furnace using a gradual heating program up to 600 °C for 16 h to destroy organic matter. Ashes were dissolved in 1.0 M HCl by heating and stirring for 1 h. A weighed aliquot of yttrium carrier (10 mg Y^{3+} mL^{-1}) was added into the completely dissolved spiked diet sample to quantify the chemical yield throughout the radiochemical separation procedure. pH was adjusted in the range of 1.0–1.2 for extracting ^{90}Y from a solution with 10 % HDEHP. After this step, yttrium was back extracted from organic phase using 3 M HNO_3. The time is recorded as the chemical separation time (t_1). Yttrium hydroxide was precipitated by adjusting the pH to 9–10 with concentrated NH_3 and centrifuged at 3000 rpm for 5 min. The supernatant was discarded and $Y(OH)_3$ precipitate was dissolved in 1 mL conc. HNO_3. 14 mL distilled water was added to the final solution and then transferred into a 20 mL plastic scintillation vial coated with Teflon. The vial was measured with Quantulus 1220 Ultra Low Level Liquid Scintillation Spectrometer for 180 min (60 min 3

cycles). A blank prepared by adding 1 mL conc. HNO_3 into 14 mL distilled water was measured before and after sample.

Cherenkov radiation produced by ^{90}Y was measured in order to determine ^{90}Sr activity concentration. The results were evaluated by using the tritium configuration of multichannel analyser of the liquid scintillation spectrometer in low coincidence bias between channels 5-320 which corresponds to the highest figure of merit. The schematic diagram of the sequential separation is shown in Figure 4.

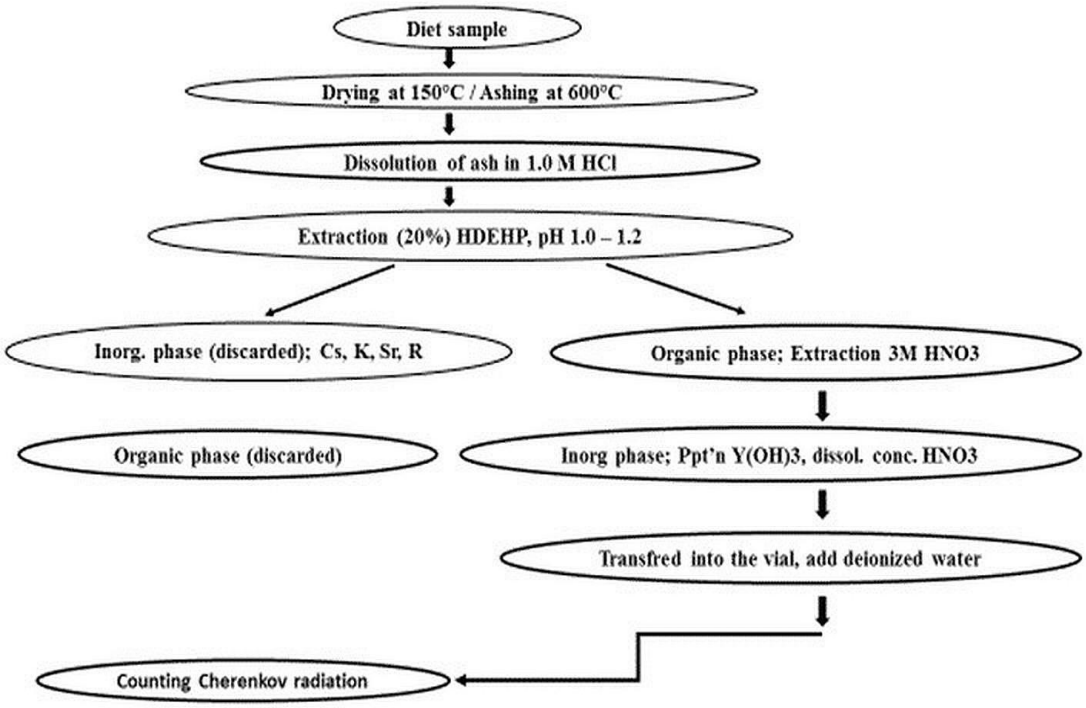

Figure 4: The schematic diagram of the Cherenkov counting method.

Determination of radiochemical recovery

The activity of ^{90}Y was measured using the calibrated liquid scintillation spectrometer (Quantulus 1220, Perkin-Elmer) in predefined and optimized energy windows with Cerenkov counting. After the measurement, the chemical yield of yttrium was determined by the complexometric titration of the sample in the scintillation vial with Titriplex III. For this purpose, aqueous sample solution in the scintillation vial, after measurement of Cherenkov radiation, was transferred into the beaker and diluted to 50 mL with ultra-pure water. Sodium acetate and xylenol orange in KNO_3 were added; pH was adjusted to 5–6 with NaOH and titrated with 0.10 M Titriplex III. The mL of titrant required to reach the endpoint were recorded as T_{smp}. The procedure was repeated with a standard by titrating the same volume of yttrium carrier as had been added to the sample during the chemical separation and extraction procedure for yttrium. The volume of titrant required for the standard was recorded as T_{std}. The chemical yield of ^{90}Y, *f*, is calculated from the ratio of T_{smp}/T_{std} where T_{std} and T_{smp} are the volumes of Titriplex III solution in mL consumed during the titration of the $^{90}Sr/^{90}Y$ standard solution and the sample, respectively.

The radiochemical recovery, R_{chem}, of the method was determined by combusting spiked diet samples with four different activity concentrations of ^{14}C and calculated by comparing the added ^{14}C activity with the measured ^{14}C activity using Eq. (1).

$$R_{chem},\% = \frac{(A_{measured,^{14}C})}{A_{added,^{14}C}} \times 100 \quad (1)$$

where $A_{measured,\ ^{14}C}$ and $A_{added,\ ^{14}C}$ are the measured and added activity of the ^{14}C (Bq) in diet samples, respectively.

Minimum detectable activity concentration (MDC)

A reagent blank sample was prepared identical to the real samples to determine the background counting rate. MDC was calculated with blank samples using the formula reported by Currie [9]:

$$MDA = \frac{\left[\left(2.71 + 4.65\sqrt{N_b}\right)\right]}{\varepsilon * m * t} \quad (2)$$

where,

N_b; is the total counts in the blank sample spectrum, cps,
t; is the counting time for the blank, in seconds,
ε; is the counting efficiency,
m; is the sample mass, in gram.

Calculation of annual effective dose

An annual effective dose was calculated according to the following equation [10]:

$$D_e = D_f \times U \times C_d \times h \quad (3)$$

Where,
D_e *;* annual effective dose in μSv.y^{-1},
D_f ; dose coefficient in μSv.Bq^{-1},
U; amount of the food consumed in 1 year in kg.y^{-1},
C_d; activity concentration of radionuclide in dried sample in Bq.kg^{-1},
h; the ratio of dried to fresh foods.
Dose coefficients (μSv.Bq^{-1}) were used for ^{90}Sr = 2.8×10^{-2}; for ^{40}K = 6.2×10^{-3}; ^{14}C; 5.8×10^{-10} (HN 73: 2003).

Results and Discussion

The main pathways of radionuclides in the human body are inhalation and ingestion through food and drinking water. Strontium-90, Caesium-134, Caesium-137 and Carbon-14 are the highest contributors to the internal dose to the people from fallout radionuclides. For this reason, there is growing interest in the radionuclides that enter the body through the diet. In this study, ^{90}Sr, ^{14}C, ^{134}Cs and ^{137}Cs activity concentrations in mixed diet samples were determined by liquid scintillation and gamma spectrometric methods.

The recovery studies for liquid scintillation spectrometry were carried out by spiked diet samples with the different, well known activity concentrations of ^{90}Sr and ^{14}C. The radiochemical recoveries were calculated by comparing the added ^{90}Sr and ^{14}C activities with the measured and results are listed in Table 1 and Table 2, respectively. As can be seen from the tables, the radiochemical recovery of the applied methods for determining the ^{90}Sr and ^{14}C radioisotopes ranged from 96% to 99% and from 85% to 102%, respectively. These results are a good indication of the validity of the method.

Table 1: ^{90}Sr recovery from the spiked diet samples

Sample	A_{Spiked}, Bq.kg^{-1}	$A_{measured}$, Bq.kg^{-1}	Relative Error, %	Recovery, %	RSD, %
Spike ^{90}Sr_1	2.226 ± 0.011	2.139 ± 0.102	4.06	96.19	6.15
Spike ^{90}Sr _2	2.008 ± 0.010	1.991 ± 0.057	0.85	99.15	1.20

Table 2: ^{14}C recovery from the spiked diet samples.

Sample	A_{spiked}, Bq.kg^{-1}	$A_{measured}$, Bq.kg^{-1}	Relative Error, %	Recovery, %	RSD, %
Spike ^{14}C_1	28.23 ± 0.88	24.06 ± 4.17	14.78	85.23	2.57
Spike ^{14}C_2	37.18 ± 1.15	34.47 ± 2.71	7.29	92.71	1.92
Spike ^{14}C_3	23.21 ± 0.72	23.51 ± 0.30	-1.31	101.29	0.21
Spike ^{14}C_4	30.38 ± 0.94	28.59 ± 1.79	5.88	94.11	1.26

The MDC value for ^{90}Sr is calculated between 0.437 Bqkg^{-1}-0.590 Bqkg^{-1} using 300 min. counting time, a background of 3 cpm, a counting efficiency of 0.98 and chemical recovery of 97 %. The MDC of ^{14}C was also found to be a 25 Bqkg^{-1} with 300 min. counting time, a background of 7 cpm, a detection efficiency of 0.65 chemical recovery of 93 % and the sample mass of about 0.5 g.

The gamma spectrometric and validated liquid scintillation spectrometric methods were applied for the determination of the activity concentration of ^{90}Sr, ^{14}C, ^{40}K, ^{134}Cs and ^{137}Cs radioisotopes in total diet samples collected in a sampling site of Mersin. Results were presented as Bq.kg^{-1} dry weight (d.w.) in Table 3 together with the uncertainty, MDC values and annual effective doses.

Natural levels of radioactivity in food are quite low and there is normally no specific legislation prescribing limits for radionuclides in food. However, in the case of a nuclear accident or radiological emergency, Council Regulation (EURATOM) 3954/87 sets maximum allowable levels of radioactivity in food- and feed-stuffs [11]. According to this regulation, maximum permitted levels of strontium isotopes (especially ^{90}Sr) and all other radionuclides of half-life greater than 10 days, notably ^{134}Cs and ^{137}Cs are 750 Bq.kg^{-1} and 1250 Bq.kg^{-1}, respectively.

All the diet samples analysed showed activity concentrations for ^{40}K varied from 282 to 486 Bq.kg^{-1}. On the other hand, the gamma emitting anthropogenic radionuclides ^{134}Cs and ^{137}Cs were not detected in any of the diet samples. MDCs are in the range of 2.4-3.3 Bq.kg^{-1} and 1.8-2.3 Bq.kg^{-1} for ^{134}Cs and ^{137}Cs respectively. These results indicate that no significiant terrestrial contamination is present in the sampling area.

Diet samples were also analysed for ^{90}Sr and ^{14}C activity concentrations and ^{90}Sr was absent in the samples taken from Yanışlı, Eceli and Gülnar sites. The detected activity concentrations of ^{90}Sr for Aydıncık and Sipahili sampling points are less than the Council Regulation (EURATOM) 3954/87 [11].
As can be seen from the Table 3, the average activity concentrations of ^{14}C were found 53, 69, 97, 34 and 107 $Bq.kg^{-1}$ d.w. in the sampling points of Yanışlı, Aydıncık, Eceli, Gülnar and Sipahili, respectively. Our results are good agrement with the average value of ^{14}C for foodstuff as reported by Saxen and Hanste [7].

Table 3: ^{90}Sr, ^{14}C, ^{40}K, ^{134}Cs and ^{137}Cs activity concentrations in mixed diet samples.

Sampling area	Radioisotope	Activity, $Bq.kg^{-1}$	*Uncertainty, $Bq.kg^{-1}$	MDC, $Bq.kg^{-1}$
Yanışlı	^{134}Cs	< MDC	-	2.4
	^{137}Cs	< MDC	-	1.8
	^{40}K	436	39	-
	^{90}Sr	< MDC	-	0.437
	^{14}C	53	4	-
Aydıncık	^{134}Cs	< MDC	-	2.7
	^{137}Cs	< MDC	-	2.3
	^{40}K	408	52	-
	^{90}Sr	1.077	0.0020	-
	^{14}C	69	7	
Eceli	^{134}Cs	< MDC	-	2.9
	^{137}Cs	< MDC	-	2.1
	^{40}K	282	48	-
	^{90}Sr	< MDC	-	0.590
	^{14}C	97	7	-
Gülnar	^{134}Cs	< MDC	-	2.4
	^{137}Cs	< MDC	-	1.8
	^{40}K	342	37	-
	^{90}Sr	< MDC	-	--
	^{14}C	34	4	
Sipahili	^{134}Cs	< MDC	-	3.3
	^{137}Cs	< MDC	-	2.3
	^{40}K	384	44	-
	^{90}Sr	1.319	0.0026	-
	^{14}C	107	10	-

**Uncertainties arising from counting statistics, background, weighing, half life, chemical recovery and efficiency were taken into account.*

Annual effective doses calculated using the results of measurements and equation (3) are presented in Table 4.

Table 4: Annual effective dose caused by ^{90}Sr, ^{14}C and ^{40}K in mixed diet.

Sampling Point	Weight of sample, kg / 3 days	Annual effective dose due to ^{90}Sr, µSv/y	Annual effective dose due to ^{14}C, µSv/y	Annual effective dose due to ^{40}K, µSv/y
Yanışlı	6.325	-	1.05	189
Aydıncık	6.550	2.11	3.69	177
Eceli	6.480	-	4.21	122
Gülnar	6.685	-	2.80	148
Sipahili	6.845	2.58	2.35	167

Internal dose conversion coefficients (µSv.Bq^{-1}): ^{90}Sr 2.8×10^{-2}; ^{40}K 6.2×10^{-3}; ^{14}C 5.8×10^{-10}

The average anual dose from ^{14}C in the mixed diet is estimated to be about 3 µSv/y. This result is consistent with the 12 µSv/y dose resulting from the natural level as presented by UNSCEAR (2000) [4]. As a result we can say that, ^{14}C concentration in Mersin is about the background level worldwide.

Conclusion

As a result of this study, a new analytical method especially for the determination of ^{14}C activity concentration in mixed diet samples using liquid scintillation spectrometer is developed and applicability of the duplicate portion technique as sampling procedure is demonstrated. The results obtained for the spiked diet samples also indicate that the recommended procedures could be successfully applied for the accurate determination of strontium-90 and carbon-14 activity concentrations in this matrix.

The data given in Table 3 confirm that activity concentrations of ^{90}Sr, ^{14}C, ^{40}K, ^{134}Cs and ^{137}Cs in mixed diet samples are low and, for the majority of samples and radionuclides, below the detection limits. They do not pose a significant risk to the human health of the population located in the sampling area.

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